

## Standard Reference Material® 70b Potassium Feldspar CERTIFICATE OF ANALYSIS

**Purpose:** This Standard Reference Material (SRM) is intended primarily for the evaluation of methods for analysis of constituent elements in feldspar or material of similar matrix.

**Description:** SRM 70b is powdered potassium feldspar that was sieved to less than 75  $\mu$ m (<200 mesh) and blended to ensure homogeneity. A unit of SRM 70b consists of one bottle containing approximately 40 grams of powder.

**Certified Values:** Certified values for elements of SRM 70b are reported in Table 1 as mass fractions on an as-received basis [1]. A NIST-certified value is a value for which NIST has the highest confidence in its accuracy, in that all known or suspected sources of bias have been investigated or taken into account [2]. A certified value is the present best estimate of the true value based on the results of analyses performed at NIST.

Table 1. Certified Mass Fraction Values for SRM 70b

| Mass Fraction |   |  |
|---------------|---|--|
|               | (%)   |  |
| 7.98          | $\pm$                                       | 0.34   |
| 0.1770        | ±   | 0.0051   |
| 6.33          | $\pm$                                       | 0.74   |
| 2.36          | $\pm$                                       | 0.15   |
|               |   |  |
| Mass Fraction |   |  |
| (             | mg/k  | g)   |
| 28.2          | ±   | 6.0  |
| 63.0          | $\pm$                                       | 4.7  |
| 790           | $\pm$                                       | 53   |
|               | 7.98<br>0.1770<br>6.33<br>2.36<br>Mas<br>(9 | (%) 7.98 ± 0.1770 ± 6.33 ± 2.36 ±  Mass Fra (mg/k) 28.2 ± 63.0 ± |

Certified Mass Fraction Values: The measurands are the mass fractions of the elements in feldspar. The certified values are metrologically traceable to the International System of Units (SI) unit of mass. Each certified value is the DerSimonian-Laird [3,4] estimate of the mean of a random effects model fitted to the data from two methods. The uncertainty listed with each certified value is an expanded uncertainty calculated according to the ISO/JCGM Guide [5,6], and it expresses contributions from all recognized sources of uncertainty, including differences between analytical methods, dispersion of values resulting from sample preparation and replicated measurement, preparation and measurement of calibrants, analytical calibration function, assay of primary materials, and balance calibration. The effective coverage factor for each evaluated uncertainty is 2, except for calcium for which it is 2.5.

Non-Certified Values: Non-certified values are provided in Appendix A.

**Additional Information:** Additional information is provided in Appendix B

**Period of Validity:** The certified values delivered by **SRM 70b** are valid within the measurement uncertainty specified until **01 September 2033**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Carlos A. Gonzalez, Chief Chemical Sciences Division Certificate of Analysis Revision History on Page 2 Steven J. Choquette, Director Office of Reference Materials

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

Use: To relate analytical determinations to the assigned values on this Certificate of Analysis, a minimum test portion of 100 mg is recommended on the basis of homogeneity testing and quantitative analyses performed at NIST. The powder does not require preparation prior to weighing because the loss on ignition at  $950 \,^{\circ}\text{C}$  is  $< 0.5 \,^{\circ}\text{M}$ .

**Storage:** The material should be stored in its original container, tightly capped, in a cool, dry location.

## REFERENCES

- [1] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; Gaithersburg MD (2008); available at https://www.nist.gov/pml/special-publication-811 (accessed Mar 2024).
- [2] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory; NIST Special Publication 260-136, 2021 edition; National Institute of Standards and Technology, Gaithersburg, MD (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed Mar 2024)
- [3] .DerSimonian, R.; Laird, N.; Meta-analysis in Clinical Trials; Control Clin. Trials, Vol. 7, pp. 177–188 (1986).
- [4] Rukhin, A.L.; Weighted Means Statistics in Interlaboratory Studies; Metrologia, Vol. 46(3), pp. 323–331, 2009.
- [5] JCGM 100:2008; Evaluation of Measurement Data Guide to the Expression of Uncertainty in Measurement; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Mar 2024); see also Taylor, B.N.; Kuyatt, C.E.; Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results; NIST Technical Note 1297, Gaithersburg MD (1994); available at https://www.nist.gov/pml/nist-technical-note-1297 (accessed Mar 2024).
- [6] JCGM 101:2008; Evaluation of Measurement Data Supplement 1 to the Guide to the Expression of Uncertainty in Measurement Propagation of Distributions Using a Monte Carlo Method; JCGM (2008); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Mar 2024)

Certificate Revision History: 26 March 2024 (Change of period of validity; updated format; editorial changes); 31 December 2013 (Original certificate date).

Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

\* \* \* \* \* \* \* End of Certificate of Analysis \* \* \* \* \* \*

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## APPENDIX A

Non-Certified Values: Non-certified values for elements are reported in Table A1 as mass fractions on an as-received basis. A non-certified value is the best estimate of the true value based on available data. These values do not meet NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement reproducibility, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [2].

The measurands are the mass fractions of elements in feldspar, as determined by the method indicated. The non-certified values are metrologically traceable to the SI unit of mass. Each non-certified value is either the mean or the median of the replicated measured values from a single method indicated in Table B2, in some cases after identification and removal of outliers. The uncertainty listed with each non-certified value is an expanded uncertainty calculated according to the ISO/JCGM Guide [5,6], and it expresses contributions from all recognized sources of uncertainty, including dispersion of values resulting from sample preparation and replicated measurement, preparation and measurement of calibrants, analytical calibration function, assay of primary materials, and balance calibration. The effective coverage factor for each evaluated uncertainty is 2, except for vanadium and zinc for which it is 2.1.

Table A1. Non-Certified Values for SRM 70b

| Element        | Mas   | ss Fr         | action |
|----------------|-------|---------------|--------|
| Iron (Fe)      | 0.13  | ±             | 0.02   |
| Silicon (Si)   | 34.4  | $\pm$         | 0.4    |
| Element        | 11100 | ss Fr<br>mg/l | action |
| Lead (Pb)      | 57    | $\pm$         | 3      |
| Magnesium (Mg) | 298   | $\pm$         | 20     |
| Rubidium (Rb)  | 495   | $\pm$         | 32     |
| Strontium (Sr) | 27    | $\pm$         | 2      |
| Titanium (Ti)  | 32    | $\pm$         | 3      |
| Vanadium (V)   | 0.93  | $\pm$         | 0.01   |
| Zinc (Zn)      | 7.7   | $\pm$         | 0.1    |

**Maintenance of Non-Certified Values:** NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this Certificate of Analysis and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

\* \* \* \* \* \* \* \* \* \* \* End of Appendix A \* \* \* \* \* \* \* \* \*

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## APPENDIX B

**Values of Potential interest:** A value for chromium is reported in Table B1 as a mass fraction on an as-received basis. This value may be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value [2].

The value reported for chromium is the mean value from one method performed at NIST.

Table B1. Value of Potential Interest for SRM 70b

| Element       | Mass Fraction |
|---------------|---------------|
|               | (mg/kg)       |
| Chromium (Cr) | 0.7           |

Table B2. Analytical Methods Performed at NIST

| Method   | Element  |
|--|--|
| X-ray fluorescence (XRF) spectrometry after borate fusion          | Al, Ba, Ca, K, Fe, Mg, Mn, Na, P, Pb, Rb, Si, Sr, Ti |
| Inductively coupled plasma optical emission spectrometry (ICP-OES) | Ba, Cr, P, V, Zn                                     |
| Instrumental neutron activation analysis (INAA)                    | Al, Ca, K, Mn, Na                                    |

**Preparation and Analysis:** The material for SRM 70b was prepared from high-purity, ceramic grade feldspar obtained from pegmatite deposits in the Custer feldspar district in the Black Hills of South Dakota. The material for SRM 70b is a mixture of alkali feldspar, plagioclase feldspar, and quartz with a small amount of mica determined using X-ray diffraction. The material was blended and bottled at NIST. Methods used by NIST for quantitative analyses are provided in Table B2.

The coordination of technical measurements for certification was performed by J.R. Sieber of the NIST Chemical Sciences Division.

Analyses leading to the certification of this SRM were performed at NIST by A.F. Marlow, D.J. O'Kelly, S.A. Rabb, and J.R. Sieber of the NIST Chemical Sciences Division and P.E. Stutzman of the NIST Materials and Structural Systems Division.

Statistical consultation for this SRM was provided by A. Possolo of the NIST Statistical Engineering Division.

Support aspects involved with the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

\* \* \* \* \* \* \* \* \* \* \* End of Appendix B \* \* \* \* \* \* \* \* \* \*

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